

QUANTITATIVE INVESTIGATIONS OF INTERFACES AND GRAIN BOUNDARIES BY PHASE CONTRAST ELECTRON MICROSCOPY WITH ULTRA HIGH RESOLUTION

C. Kisielowski*, J.M. Plitzko**, S. Lartigue***, T. Radetic*, U. Dahmen*

*National Center for Electron Microscopy, Ernest Orlando Lawrence Berkeley National Laboratory, University of California, Berkeley, CA 94720

**Lawrence Livermore National Laboratory, P.O. Box 808, Livermore, CA 94551

*** CECM Vitry, CRNS, France

Recent progress in High Resolution Transmission Electron Microscopy makes it possible to investigate crystalline materials by phase contrast microscopy with a resolution close to the 80 pm information limit of a 300 kV field emission microscope¹⁻⁴). A reconstruction of the electron exit wave from a focal series of lattice images converts the recorded information into interpretable resolution^{5,6}). The present contribution illustrates some recent applications of this technique to interfaces.

Fig. 1 shows a reconstructed electron exit wave of a heterophase interface between GaN and sapphire. The experiment takes advantage of three factors: First, we resolved the GaN lattice in [1 $\bar{1}$ 00] projection, which requires at least 0.15 nm resolution. The [1 $\bar{1}$ 00] projection eliminates the stacking fault contrast that usually obscures lattice images in the commonly recorded [11 $\bar{2}$ 0] projection. Thus, image interpretation is drastically simplified. Second, all atom columns at the interface and in the sapphire are resolvable with a smallest projected aluminum - oxygen spacing of 85 pm in the sapphire. Consequently, it is now possible to detect single columns of oxygen at a sub Angstrom spacing because of the excellent signal to noise ratio of the phase contrast image. Third, the reconstructed phase image is directly interpretable because a bright spot in the electron exit wave marks the position of atomic columns. The reconstruction procedure is applicable to any crystalline material. Figures 2 ⁷⁾ and 3 illustrate further examples of this technique to grain boundaries in metals and oxides.

Current efforts aim for a quantification of the information from the reconstructed exit waves. It has already been demonstrated that displacements of the atomic columns as small as 1 –3 pm can be extracted from lattice images or from the reconstructed electron exit wave ^{8,9)}. Furthermore, local thickness and chemical composition can in principle be accessed through the magnitude of the phase change¹⁰⁾. We tested the possibility of determining the number of atoms in [110] columns of gold, which is a suitable material because of its short extinction distance. From simulations (Figure 4a) one expects a maximum phase change of π in a column of only 9 Au atoms. Figure 4b depicts an experimental line trace across single columns of Au atoms in a reconstructed phase image from a wedge shaped sample. The largest recorded phase change in the experiment was normalized to π . It is seen that one would in fact count 8-9 Au atoms to reach the maximum. Therefore, it is feasible to detect the phase change of single gold atoms by quantitative analyses of the electron exit wave. ¹¹⁾ In addition to demonstrating the current level of instrument performance, future needs and opportunities for quantitative HRTEM of interfaces will be discussed.

References:

1. C. Kisielowski, E.C. Nelson, C. Song, R. Kilaas, A. Thust, *Microscopy and Microanalysis* 6, 2000, 16
2. M.A. O'Keefe, *Microscopy and Microanalysis* 6, 2000, 1192
3. C. Kisielowski, C.J.D. Hetherington, Y.C. Wang, R. Kilaas, M.A. O'Keefe, A. Thust, *Sub. to Ultramicroscopy*, 2000
4. M.A. O'Keefe, C.J.D. Hetherington, Y.C. Wang, E.C. Nelson, J.H. Turner, C. Kisielowski, J.-O. Malm, R. Mueller, J. Ringnalda, M. Pam, A. Thust, *Sub. to Ultramicroscopy*, 2000
5. W.M.J. Coene, A. Thust, M. Op de Beeck, D. Van Dyck, *Ultramicroscopy* 64, 1996, 109
6. A. Thust, W.M.J. Coene, M. Op de Beeck, D. Van Dyck, *Ultramicroscopy* 64, 1996, 211
7. J.M. Plitzko, G.H. Campbell, W.E. King, S.M. Foiles in J. Bentley, U. Dahmen, C. Allen, I. Petrov, (eds) *Materials Research Society Symp.* **589**, 2000, in press
8. C. Kisielowski, O. Schmidt, J. Yang, *Mat. Res. Soc. Symp.*, **482**, 1998, 369
9. C.L. Jia, A. Thust, *Phys. Rev. Lett.* 82, 1999, 5052
10. D. van Dyck, J.H. Chen, *Solid State Communications* 109, 1999, 501

11. The project is sponsored by the Director, Office of Science, Office of Basic Energy Sciences, of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor The Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or The Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof, or The Regents of the University of California. Ernest Orlando Lawrence Berkeley National Laboratory is an equal opportunity employer.